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TECHNICAL MEMORANDUM 1091

EVALUATION OF DOPED PERCHLORATES IN
EXPERIMENTAL PHOTOFLASH COMPOSITIONS

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OCTOBER 1963

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**EVALUATION OF DOPED PERCHLORATES IN
EXPERIMENTAL PHOTOFLASH COMPOSITIONS**

by

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October 1963

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TABLE OF CONTENTS

	Page
Object	1
Summary	1
Introduction	2
Results	3
Discussion of Results	3
Conclusions	8
Recommendations	9
Experimental Procedures	10
Preparation of Materials	10
Blending and Loading Procedures	10
Testing	10
Materials	10
References	11
Distribution List	18
Table	
1 Luminosity Characteristics of Experimental Photo- flash Systems	12
2 Luminosity Characteristics of Experimental Photo- flash Systems	13
3 Luminosity Characteristics of Experimental Photo- flash Systems	14
4 Comparison of Average Efficiency of Doped Perchlorate Systems at Sea Level and 80,000 Feet Simulated Altitude	15

	Page
Table	
5 Impact and Friction Sensitivity Data	16
Figure	
1 Average time-intensity curves at sea level and simulated high altitude	17

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OBJECT

To evaluate the effects upon luminosity produced by introducing the impurities Ag^+ , Cu^{++} , and I^- ions into the crystal lattice of potassium perchlorate when this compound is incorporated into a high energy fuel/oxidant flash system.

SUMMARY

The effects of an oxidant containing added impurities such as Ag^+ , Cu^{++} , and I^- ions upon the luminosity of flash systems were tested in the following blends:

1. FP600 (60/40 potassium perchlorate/aluminum) photoflash composition for sea level applications
2. FP790 (30/20/50) calcium/aluminum/potassium perchlorate) photoflash composition for high altitude applications
3. FP856 (31/20/49 aluminum/calcium fluoride/potassium perchlorate) high altitude flash composition

All tests were performed at sea level or at 20.9 mm Hg, simulating 80,000 feet altitude,* and at the temperature prevailing at the Pyrotechnic Laboratory High Altitude Tank.

The system 40/60 aluminum/potassium perchlorate doped with Ag^+ , Cu^{++} , or I^- ions exhibited no increase in luminous efficiency over a non-doped control at ambient conditions. At 80,000 feet, increased efficiencies were achieved with the doped systems, but the results could not be verified in a second series of tests.

Within the systems 30/20/50 calcium/aluminum/potassium perchlorate and 31/20/49 aluminum/calcium fluoride/potassium perchlorate, the compositions containing potassium perchlorate doped with Ag^+ ions showed the greatest increases in luminous efficiencies, ranging up to 27.0% and 29.4% respectively at 80,000 ft. However, neither system gave verifiable results on retest when the method of doping was stepped up from laboratory to pilot plant scale.

The above systems doped with Cu^{++} and I^- ions gave results that were either not verifiable or else exhibited lower efficiency values. In the case of the Cu^{++} ion, which was introduced as the perchlorate, there was an undesirable reaction between its water of hydration and calcium.

* In this report the terms "20.9 mm Hg" and "80,000 feet" are used interchangeably. All altitudes are simulated, not actual.

Increasing the concentration of Ag^+ ions from 1 to 2 mole percent did not improve the luminous efficiencies of the systems in which they were used.

INTRODUCTION

The program outlined in this report was suggested by the results obtained by Freeman et al (Refs 1, 2, and 3) working with irradiated and non-irradiated samples of ammonium perchlorate and samples of doped and undoped potassium perchlorate in combination with certain fuels (Ref 4). It was found that nonirradiated potassium perchlorate decomposes at lower temperatures in the presence of magnesium and aluminum if the potassium perchlorate is not pure but contains small quantities of silver, cupric, or iodide ions. In studies of a blend of 20/80 magnesium/potassium perchlorate the ignition temperature was reduced from 646°C to 500°C when the potassium perchlorate was doped with one mole percent silver perchlorate. The time to ignition for the silver doped composition was approximately half of that obtained with the nondoped or pure system (Ref 5). In 40/60 aluminum/potassium perchlorate, decomposition of the potassium perchlorate (as determined by both TGA and DTA techniques) began at 580°C . When doped with one mole percent silver perchlorate, the potassium perchlorate started to decompose at 508°C , but did not ignite. In both cases, it was postulated that the silver ion provides nuclei for reaction. It was also found that, in the presence of cupric and iodide ions, the decomposition of both ammonium and potassium perchlorate is considerably enhanced. This finding was attributed to the low electron affinity and polarizability of the iodide ion and to the ability of the cupric ion to act as an electron bridge in an electron transfer mechanism of decomposition. In view of the profound changes in the observed ignition temperatures due to the above impurities, it was considered desirable to determine whether the performance characteristics of flash compositions containing potassium perchlorate may be altered by doping the potassium perchlorate with these cationic and anionic impurities.

The purpose of the present investigation was to determine whether there is a relationship between the reactivity of doped systems and the luminosity results obtained at both sea level and simulated high altitudes.

RESULTS

Tables 1 through 3 (pp 12 through 14) contain all the parameters measured to determine the average luminous efficiencies, in candleseconds per gram, of the photoflash systems tested. In each group the upper figures are average values and the lower figures are the range of values.

Table 1 (Nos. 1-4), p 12, lists the CIE results for the 40/60 aluminum/potassium perchlorate system. All doped perchlorates used in these tests were prepared on a laboratory scale in one-pound lots and test fired during June 1960.

Table 2 (p 13) lists the CIE values for confirmatory tests with the 40/60 aluminum/potassium perchlorate system containing laboratory prepared doped perchlorates (Nos. 5-8). This table also shows additional values obtained for FP 790 and FP 856 systems containing calcium and calcium fluoride (Nos. 9-15). These items were tested in March 1961.

Table 3 (Nos. 16-23) lists the CIE values obtained for the FP 790 and FP 856 systems, and their doped variants. This table differs from Table 2 in that the doped perchlorates were prepared in five-pound lots on a pilot plant scale. These items were fired in September 1961.

Table 4 (p 15) compares the average efficiencies of all the systems tested, showing the percentage increase of each system in going from ambient to 80,000 feet, and giving the percentage increase relative to the control system at both ambient and 80,000 feet.

Table 5 (p 16) lists the friction pendulum and impact sensitivity results for the systems under investigation.

Figure 1 (p 17) depicts typical average time vs luminous intensity curves derived at both sea level and 80,000 feet simulated altitude for the systems evaluated.

DISCUSSION OF RESULTS

For purposes of discussion, only the average efficiencies need be referred to. Hence, Table 4 will be reviewed in detail.

The luminosity results given in Table 4 for the 40/60 aluminum/potassium perchlorate composition (Nos. 1-4) were obtained with doped perchlorates

that were prepared on a laboratory scale and tested during June 1960. An examination of the total average efficiencies at 80,000 feet reveals higher values for the doped systems when compared to their controls, e.g., 5,600, 5,600, and 4,400 candleseconds/gram for silver perchlorate, cupric perchlorate, and potassium iodide, respectively, against 4,300 candleseconds/gram for the nondoped control. The ranges, however, reveal considerable overlap with the control (Table I, Nos. 1-4, p 12). At sea level, there were no increases in efficiency over the 7,320 candleseconds/gram value obtained with the control system. It should be mentioned at this point that the total efficiency values and not the 1/20 maximum efficiency values were examined. This was done because subsequent data was obtained by means of an electronic integrator that computed only the total area under the CIE curve. Table 4 (p 15) also shows the percentage increases or decreases obtained in the 60/40 system. The silver and copper doped perchlorates (Nos. 2 and 3) show 25.0 and 30.0% increases over the control at 80,000 feet. The potassium iodide doped system (No. 4) yielded only a 2.3% increase.

To verify the percentage increases achieved with the 40/60 aluminum/potassium perchlorate system and to test doped perchlorates with two other systems, FP 790 (30/20/50 calcium/aluminum/potassium perchlorate) and FP 856 (31/20/49 aluminum/calcium fluoride/potassium perchlorate), new batches of doped perchlorates were prepared on a laboratory scale. Table 4 (Nos. 5-15) contains efficiency data on these systems obtained during March 1961 at sea level and 80,000 feet.

The 60/40 potassium perchlorate/aluminum compositions containing oxidant doped with Ag^+ , Ca^{++} , and I^- ions (Nos. 6, 7, 8) exhibited little or no increase in luminosity over a nondoped control system when tested at ambient conditions. At 80,000 feet simulated altitude, the doped system again showed no increase over the control, and all systems gave lower values in light output at high altitude than corresponding systems fired under sea level conditions.

The FP 790 system (30/20/50 calcium/aluminum/potassium perchlorate) exhibited increases in luminosity over the control when an oxidant doped with 1 mole percent silver perchlorate was used. The average efficiency of the control (No. 9) was 5200 candleseconds/gram at sea level and 11,600 candleseconds/gram at 80,000 feet. The latter figure represents an increase of 122% over sea level. For the FP 790 system containing potassium perchlorate doped with 1 mole percent silver perchlorate (No. 10),

the efficiencies obtained were 8,200 candleseconds/gram at sea level, and 14,700 candleseconds/gram at 80,000 feet, showing an increase of 79% over sea level. This system, however, exhibited an efficiency increase of 58% over the control at sea level and of 27% over the control at 80,000 feet. In this series of tests it was noted that the FP 790 silver perchlorate doped system was superior in sea level efficiency to the standard 60/40 potassium perchlorate/aluminum system (8,200 and 5,200 candleseconds/gram, respectively). This is a reversal of past observation where the performance of the 60/40 potassium perchlorate/aluminum at sea level has always been superior to that of a nondoped FP 790 system. The potassium iodide doped system (No. 11) showed no increase over the control at either sea level or altitude. The use of cupric perchlorate doped potassium perchlorate in the FP 790 composition was dropped because of reactivity between metallic calcium and the hydrated cupric perchlorate.

The control system FP 883 (31/20/49 aluminum/calcium fluoride/potassium perchlorate) gave efficiencies of 2,200 and 4,700 candleseconds/gram at sea level and high altitude, respectively (Table 4, No. 12). This represents an increase of 112% at high altitude. This system doped with 1 mole percent silver perchlorate (No. 13) gave efficiencies of 2,800 and 6,000 candleseconds/gram at sea level and 80,000 feet, respectively. The 1,800 candleseconds/gram figure represents a decrease in efficiency at sea level when compared with the control, but 6,000 candleseconds/gram at 80,000 feet represents a 27% increase over the high altitude control due to the presence of the dopant, and a 232% increase over the sea level value of 1,800 candleseconds/gram.

The two other doped oxidant systems evaluated in the FP 883 system, cupric perchlorate doped potassium perchlorate (No. 14) and potassium iodide doped potassium perchlorate (No. 15), proved either erratic in performance or were not significantly more efficient than their respective controls.

To obtain additional quantities of doped perchlorates for the purpose of substantiating past results and in anticipation of larger scale production, 5-pound batches of doped potassium perchlorate were prepared on a plant scale and additional smaller batches of doped perchlorates were prepared on a laboratory scale. It was found that the different preparations varied in average particle size. For example, laboratory recrystallized potassium perchlorate was 60 microns in average particle size. The same material after doping was about 82 microns. However, the second laboratory

preparation yielded 94-micron recrystallized potassium perchlorate and 82-micron silver-doped potassium perchlorate. The plant-produced recrystallized potassium perchlorate and the corresponding doped materials ranged from 86 to 120 microns. All preparations, consequently, were screened and ball milled to a particle size of approximately 50 microns to insure constant density during loading of the Poppy cartridges. Table 4 (p 15) lists the results obtained during September 1961 with the plant preparations (Nos. 16-23). Two additional systems, one designed to test the effect of doubling the silver perchlorate dopant concentration to 2 mole percent (Nos. 19 and 23) and the other to test the effect produced upon the system by the exposure of the silver perchlorate doped perchlorate to light for eight hours (Nos. 18 and 22), are also listed. The light exposure should result in the reduction of silver ions to metallic silver.

Considering the system FP 790 (30/20/50 calcium/aluminum/potassium perchlorate) first, it is seen that the 1 mole percent silver perchlorate doped system (No. 17) gave increases over the control of 8.5% at sea level and 3.2% at 80,000 feet. These results do not agree with the 58% and 27% increases over the respective controls previously obtained with this system (No. 10). The light-exposed system doped with 1 mole percent silver perchlorate (No. 18) yielded increases of 8.5% and 10.7% over the respective controls at sea level and high altitude. The system doped with 2 mole percent silver perchlorate (No. 19) showed no efficiency increase over controls at either sea level or high altitude.

An examination of the FP 883 (31/20/49 aluminum/calcium fluoride/potassium perchlorate) composition data shows that the system doped with 1 mole percent silver perchlorate (No. 21) failed to give sufficient light to record. These results did not agree with the previous evaluation of this system (No. 13) in which the efficiencies averaged 6,000 candleseconds/gram at 80,000 feet, an increase of 27% over the control. The light-exposed system doped with 1 mole percent silver perchlorate (No. 22) gave results which closely paralleled those previously obtained for the same non-light-exposed composition (No. 13), with an increase at high altitude of 29% over the control. As with the FP 790 composition, the system doped with 2 mole percent silver perchlorate (No. 23) showed no efficiency increase over the controls at either sea level or high altitude.

Sensitivity to impact and friction was determined for the doped and non-doped systems but no definite relationship between sensitivity and doping was apparent (Table 5, p 16).

Figure 1 (p 17) shows the configuration of some average time-intensity curves derived from the initiation of laboratory blends of doped perchlorate systems and their controls in the Poppy cartridge. Because different calibration factors are used in determining peak intensities, these curves cannot be directly compared. However, by reference to Tables 1-3 (pp 12-14), the average times to peak, peak intensities, integral light values, and durations may be found.

Examination of the time-intensity profiles for the systems studied reveals the following:

40/60 aluminum/potassium perchlorate. The peak is greater and duration longer at 80,000 feet simulated altitude. The integral light, however, is greater at sea level because the curve is broader at peak intensity. It can be assumed that at 80,000 feet the cooling rate is faster and there is a steep drop in intensity before the curve gradually levels off.

40/60 aluminum/potassium perchlorate, 1 mole percent silver perchlorate doped. At sea level, this system reveals a broad curve. At 80,000 feet, there is a sharp peak followed by rapid cooling. Again the broad curve at sea level offsets the longer duration at 80,000 feet to give greater integral light.

40/60 aluminum/potassium perchlorate, 1 mole percent cupric perchlorate doped. Although the duration is somewhat greater at 80,000 feet, there is a broader curve at sea level as well as a higher peak and consequently a greater integral light.

40/60 aluminum/potassium perchlorate, 1 mole percent potassium iodide doped. The peak intensities are comparable at sea level and 80,000 feet. At sea level there is a broader curve than at 80,000 feet, where cooling is depicted by a sharp drop in the curve. The integral light is greater at sea level than at 80,000 feet.

30/20/50 calcium/aluminum/potassium perchlorate. The integral light for this system is greater at 80,000 feet than at sea level. The broad curve at sea level does not compensate for the high peak and longer duration at 80,000 feet. Again, at high altitude, there is rapid cooling and gradual tapering off.

30/20/50 calcium/aluminum/potassium perchlorate, 1 mole percent silver perchlorate doped. This system has a greater peak intensity, duration,

and integral light at 80,000 feet than it does at sea level. These values are greater than those for the nondoped control previously described.

30/20/50 calcium/aluminum/potassium perchlorate, 1 mole percent potassium iodide doped. There is a broad curve at sea level but the peak intensity and duration at 80,000 feet account for a greater integral light. There is rapid cooling of very short duration followed by gradual tapering off.

31/20/49 aluminum/calcium fluoride/potassium perchlorate. Because of higher peak intensity and longer duration, the integral light is greater at 80,000 feet. There are no sharp peaks at either elevation.

31/20/49 aluminum/calcium fluoride/potassium perchlorate, 1 mole percent silver perchlorate doped. This system has a higher peak intensity, duration, and integral light at 80,000 feet than it does at sea level. These values are also greater than corresponding values for the control. Again, no sharp peaks are present.

31/20/49 aluminum/calcium fluoride/potassium perchlorate, 1 mole percent cupric perchlorate doped. This system was very erratic at 80,000 feet. The one curve obtained at altitude tended to peak sharply.

31/20/49 aluminum/calcium fluoride/potassium perchlorate, 1 mole percent potassium iodide doped. The configuration of both curves at sea level and 80,000 feet is similar. However, an examination of the peak intensity, duration, and integral light reveals that these values are greater at 80,000 feet.

CONCLUSIONS

1. It can be hypothesized that new nucleation sites formed by impurities affect the rate-governing factors for solid-solid reactions.
2. The purity of materials and the manner in which the dopant is introduced into the oxidant seem to be factors controlling the luminosity output of Ag^+ ion doped perchlorates, particularly at 80,000 feet. This is indicated by the results obtained with both laboratory and plant scale preparations (e.g., 100-gram and 5-pound lots). In one instance, laboratory

preparations for the 60/40 potassium perchlorate/aluminum in which the oxidant was doped with Ag^+ ion gave luminosity increases of 25% when compared to the control at 80,000 feet. However, this finding could not be duplicated in a second experiment.

3. The systems containing the light-exposed 1 mole percent silver perchlorate were more efficient at 80,000 feet than systems containing non-light-exposed doped oxidant. The low order performance of the aluminum/calcium fluoride/potassium perchlorate doped with 1 mole percent silver perchlorate as well as the considerably poorer luminosity data obtained on the retest of the similarly doped calcium/aluminum/potassium perchlorate system cannot at present be explained.

4. Sensitivity to impact and friction for the doped systems did not follow any set pattern. Ignition temperature values, however, are reported to be lower for the doped perchlorates (Ref 3).

5. The usual trend of the superior luminosity characteristics of 60/40 potassium perchlorate/aluminum when compared to the FP 790 system (30/20/50 calcium/aluminum/potassium perchlorate) at sea level was reversed when Ag^+ ion doped perchlorate was used (5,900 to 7,300 and 8,200 candleseconds/gram, respectively).

6. Evidently a critical concentration is necessary for doping perchlorates. A silver ion concentration derived from 2 mole percent silver perchlorate proves less efficient than a concentration from 1 mole percent silver perchlorate.

RECOMMENDATIONS

It is recommended that:

1. The evaluation of dopants be continued in flash and flare compositions. In addition, the effects of aging, particle size, and particle surface should be determined.

2. The activation energies for doped and nondoped perchlorates be determined and correlated, if possible, with luminous outputs in flash systems.

EXPERIMENTAL PROCEDURES

Preparation of Materials

Potassium perchlorate (1.0 g/100 cc) was dissolved in distilled water at 100°C. The solution was filtered while hot through fine filter paper and the funnel was kept warm by means of a heating mantle. The filtrate was then placed in an ice bath and precipitated with constant stirring. The supernatant was then decanted, and the recrystallized perchlorate was air dried for one hour and oven dried overnight at 100–110°C.

The recrystallized potassium perchlorate was then dissolved in distilled water and heated to 100°C. One mole percent of the doping material was introduced into the solution with stirring and the solution was then evaporated to dryness. After drying overnight at 100–110°C the salt was gently ground and stored in brown bottles. Where necessary, particle sizes were determined and adjusted to 50 microns by ball milling.

Blending and Loading Procedures

Compositions were dry blended and loaded into Poppy cartridges in accordance with sequence of operation PACU No. 5, September 1957, and all cartridges were loaded remotely in accordance with Loading Branch Sequence T 1024-2-11-5. Each cartridge contained a 200-mg lead azide charge at the base of a short delay column consisting of 90/10 barium chromate/boron. Initiation was produced by means of a squib.

Testing

All cartridges were tested in the Pyrotechnics Laboratory's simulated high altitude chamber. The items were fired at right angle to the photocell. An oscilloscope with a high-speed camera attachment was used to record the photocell output as a function of time. An integrator was used to compute the integral light.

Materials

The following materials were used:

1. Calcium, atomized, 36 micron, Valley Metallurgical Processing Company
2. Potassium perchlorate, reagent grade, Fisher Scientific Company

3. Aluminum, atomized, 15 micron, Allied Chemical Company
4. Poppy photoflash cartridge, Drawing number CX^P-111207, December 8, 1959
5. Silver perchlorate (anhydrous), G. F. Smith Chemical Company
6. Potassium iodide (Fisher Certified, A.C.S.), Fisher Scientific Company
7. Cupric perchlorate (hydrated) (Fisher Certified), Fisher Scientific Company
8. Calcium fluoride, reagent grade, J. T. Baker Chemical Company.

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TABLE 1
Luminosity Characteristics of Experimental Photoflash Systems
(Laboratory preparations tested in the Peppy cartridge during June 1960)

Composition	Weight of Composition, g	Altitude, ft	Time to Peak, m sec	Peak, 10 ⁶ candles	Total Integral Light x 10 ³ candle-sec	Total Duration, m sec	Average Efficiency, 10 ³ candleseconds/ gram
1. 40/60 aluminum/ potassium perchlorate recrystallized	21	Sea level	0.78 0.3-1.7*	33.0 31-35	153 141-177	14.7 13.8-15.1	7.3 6.7-8.4
		80,000	0.46 0.3-0.5	44.6 42.0-49.0	91.0 67.9-140.0	15.3 13.5-17.5	4.3 3.2-6.7
2. 40/60 aluminum/ potassium perchlorate + 1 mole % silver per- chlorate	18.6	Sea level	0.55 0.3-0.8	31.2 26.0-36.0	135.0 105.0-170.0	13.8 11.8-16.3	7.3 5.5-9.0
		80,000	0.38 0.3-0.5	39.4 35.0-42.0	100.4 82.0-116.0	16.4 10.3-20.0	5.4 4.4-6.2
3. 40/60 aluminum/ potassium perchlorate + 1 mole % cupric perchlorate	17.1	Sea level	1.1 0.7-2.5	27.4 22.1-32.4	120.0 92.0-144.0	14.5 9.0-17.7	7.0 5.6-8.5
		80,000	0.35 0.3-0.5	29.5 24.2-32.5	97.0 67.0-122.0	24.5 13.0-38.0	5.6 4.0-7.0
4. 40/60 aluminum/ potassium perchlorate + 1 mole % potassium iodide	23.4	Sea level	1.6 0.7-2.3	30.7 28.0-35.0	167.0 145.0-206.0	18.9 13.2-23.5	7.3 6.2-8.8
		80,000	0.83 0.7-1.0	20.5 18.7-22.0	102.0 92.0-120.0	26.5 20.0-31.0	4.4 3.9-5.2

* Range of values

TABLE 2
Luminescence Characteristics of Experimental Pin-offish Systems
(Laboratory preparations tested in the Poppy cartridge during March 1961)

Composition	Weight of Composition, g	Altitude, ft	Time to Peak, m sec	Peak, 10 ³ cond/sec	Total Integrated Light = 10 ³ cond/sec	Total Duration, m sec	Average Efficiency, 10 ³ cond/sec/g
5. 40/60 aluminum/potassium perchlorate recrystallized	21.0	Sea level	1.2 0.5-1.9	22.7 21.0-23.5	126.0 105.0-141.0	14.2 13.0-16.5	5.9 5.2-7.0
		80,000	0.5 0.3-0.8	26.3 19.1-33.0	131.0 77.0-110.0	19.5 15.5-24.5	4.8 3.7-5.3
6. 40/60 aluminum/potassium perchlorate + 1 mole % silver perchlorate	18.6	Sea level	1.3 0.8-1.5	17.1 12.0-23.0	81.0 36.6-101.2	13.5 9.5-18.0	4.4 3.0-5.4
		80,000	0.5 0.3-0.6	17.8 12.5-23.0	78.0 63.6-99.6	17.0 13.3-19.5	4.2 3.4-5.4
7. 40/60 aluminum/potassium perchlorate + 1 mole % cupric perchlorate	17.1	Sea level	1.1 0.8-1.6	12.9 9.2-16.3	37.0 36.0-74.5	12.7 1. - 11	3.34 2.2-4.4
		80,000	0.6 0.3-0.5	14.6 8.7-12.9	49.0 32.2-75.4	19.3 16.0-21.5	2.9 1.8-4.4
8. 40/60 aluminum/potassium perchlorate + 1 mole % potassium iodide	23.4	Sea level	2.2 1.5-2.5	24.2 21.5-27.5	146.0 131.0-162.0	14.9 12.5-17.0	6.2 5.6-6.8
		80,000	0.86 0.3-1.1	22.6 17.6-1.1	113 111.0-124.0	21.3 18.3-23.0	4.9 4.7-5.3
9. 50/20/50 calcium/aluminum/potassium perchlorate	17	Sea level	1.9 1.3-3.0	35.0 21.2-49.2	80.0 72.0-109.0	14.1 12.7-15.5	5.2 4.2-6.4
		80,000	0.7 0.3-1.0	35.7 34.0-40.0	197.0 163.0-262.0	22.6 23.3-25.0	11.6 9.6-15.4
10. 50/20/50 calcium/aluminum/potassium perchlorate + 1 mole % silver perchlorate	19	Sea level	2.1 1.8-2.5	21.4 21.4-29.8	146.0 127.0-298.0	15.8 14.0-16.3	8.2 6.7-10.5
		80,000	0.8 0.3-1.0	37.5 34.7-49.0	280.0 260-315.0	23.1 19.0-27.5	14.7 13.7-16.6
11. 50/20/50 calcium/aluminum/potassium perchlorate + 1 mole % potassium iodide	26	Sea level	2.3 2.0-2.5	29.7 19.5-27.2	148.0 132.0-134.5	15.7 14.3-17.0	4.9 4.3-5.6
		80,000	1.2 1.0-1.3	34.2 26.4-32.8	242.0 199.0-301.0	24.2 18.0-28.0	12.5 8.3-12.5
12. 31/20/49 aluminum/calcium fluorite/potassium perchlorate	23.1	Sea level	1.1 0.8-1.5	9.0 6.2-12.7	45.0 34.0-58.5	7.9 6.3-12.3	2.2 1.7-2.9
		80,000	1.3 1.0-1.5	13.9 12.4-14.8	94.0 76.9-113.0	18.4 13.8-23.5	4.7 3.8-5.6
13. 31/20/49 aluminum/calcium fluorite/potassium perchlorate + 1 mole % silver perchlorate	17.7	Sea level	1.5 1.0-1.7	7.0 5.8-7.8	31.5 23.0-39.0	12.2 6.0-12.5	1.8 1.3-2.2
		80,000	1.8 1.0-2.0	14.7 13.5-16.7	126 95.0-118.0	18.8 18.5-21.0	4.0 3.3-6.7
14. 31/20/49 aluminum/calcium fluorite/potassium perchlorate + 1 mole % cupric perchlorate	16.8	Sea level	1.6 1.2-2.1	7.3 6.6-8.7	31.0 14.1-41.8	8.2 6.0-9.7	1.8 0.8-2.5
		80,000	Erratic	-	-	-	-
15. 31/20/49 aluminum/calcium fluorite/potassium perchlorate + 1 mole % potassium iodide	18.5	Sea level	1.8 1.5-2.0	6.8 4.0-9.2	35.0 27.0-48.5	13.0 7.5-16.0	1.9 1.5-2.7
		80,000	2.6 1.2-4.7	11.8 11.5-13.2	90.0 73.0-105.0	19.9 17.2-22.0	5.0 4.0-5.8

TABLE 3
Luminosity Characteristics of Experimental Photoflash Systems
(Pilot-scale preparations tested in the Poppy cartridge during Sept 1961)

Composition	Weight of Composition, g	Altitude, ft	Time to Peak, μ sec	Peak, 10^6 candle/s	Total Integrated Light = 10^6 candle-sec	Total Duration, μ sec	Average Efficiency, 10^6 candle-sec/g
16. 30/20/50 calcium/aluminum/potassium perchlorate	18.7	Sea level	2.3 1.5-3.3	16.8 12.7-19.5	136.5 96.5-117.5	15.3 13.5-17.5	5.9 4.9-6.5
		80,000	1.2 0.8-1.5	39.4 28.5-32.5	224.6 192.5-260.5	26.0 24.5-28.5	12.5 10.7-14.7
17. 30/20/50 calcium/aluminum/potassium perchlorate + 1 mole % silver perchlorate	17.5	Sea level	2.2 1.4-3.0	17.5 14.4-19.5	111.9 101.0-118.5	17.5 14.5-23.0	6.4 5.8-6.9
		80,000	1.9 0.6-1.4	28.9 27.8-39.6	126.0 107.5-234.5	26.5 25.5-29.0	12.9 10.7-15.4
18. 30/20/50 calcium/aluminum/potassium perchlorate 1 mole % silver perchlorate (exposed)	17.0	Sea level	2.2 1.2-3.2	17.6 14.5-21.1	138.2 93.0-155.5	16.3 15.0-18.5	6.4 5.5-6.8
		80,000	0.9 0.7-1.1	31.2 29.8-35.6	238.5 200.5-255.5	29.2 26.0-32.5	14.5 12.2-15.5
19. 30/20/50 calcium/aluminum/potassium perchlorate 2 mole % silver perchlorate	17.5	Sea level	2.1 1.5-3.1	16.5 13.8-18.7	95.1- 87.0-101.0	14.9 13.5-16.5	5.8 5.5-13.5
		80,000	0.8 0.7-1.0	28.3 27.5-33.5	211.8 168.8-233.5	25.8 23.5-29.5	15.4 13.7-16.5
20. 31/20/49 aluminum/calcium fluoride/potassium perchlorate	18.5	Sea level	2.8 2.0-4.7	13.5 7.9-11.9	47.3 29.8-68.9	13.3 9.0-12.5	2.9 1.6-2.5
		80,000	1.5 1.5-1.6	11.7 11.0-12.4	95.0 71.5-118.5	22.2 21.5-23.5	5.1 3.9-6.1
21. 31/20/49 aluminum/calcium fluoride/potassium perchlorate, 1 mole % silver perchlorate	16.0	Sea level	Erratic				
		80,000	Erratic				
22. 31/20/49 aluminum/calcium fluoride/potassium perchlorate, 1 mole % silver perchlorate (exposed)	17.5	Sea level	2.2 2.0-2.5	9.1 4.1-13.4	32.0 25.0-17.5	10.0 7.0-12.0	1.8 * 1.5-2.1
		80,000	1.6 1.5-1.6	13.0 12.5-13.5	116.3 90.0-137.5	25.0 23.5-27.5	6.6 5.1-7.8
23. 31/20/49 aluminum/calcium fluoride/potassium perchlorate, 2 mole % silver perchlorate	18.5	Sea level	2.4 2.0-3.5	9.5 7.7-13.7	34.5 32.8-35.0	13.7 13.5-11.5	1.0 1.8-2.0
		80,000	1.7 1.5-2.0	1.2 10.8-13.5	93.0 66.5-157.5	25.5 18.0-33.0	5.3 3.8-5.8

TABLE 4
Comparison of Average Efficiency of Doped Perchlorate Systems at Sea Level and 80,000 Feet Simulated Altitude

Test Dates	Composition	Percent Comp	Average Efficiency, 10^3 candlepower/g		Percent Increase at 80,000 ft Over Sea Level	Percent Increase at Sea Level Over Control at Sea Level	Percent Increase at 80,000 ft Over Control at 80,000 ft
			Sea Level	80,000 ft			
DOPED PERCHLORATES PREPARED ON LABORATORY SCALE							
June 1960	1. Aluminum/potassium perchlorate (control)	60/60	7.3	4.3	0	-	-
	2. Aluminum/potassium perchlorate - 1 mole % silver perchlorate	60/60	7.2	5.4	0	0	25.6
	3. Aluminum/potassium perchlorate - 1 mole % cupric perchlorate	60/60	7.3	5.6	0	0	30.6
	4. Aluminum/potassium perchlorate - 1 mole % potassium iodide	60/60	7.3	6.4	0	0	2.3
March 1961	5. Aluminum/potassium perchlorate (control)	60/60	5.9	4.8	0	-	-
	6. Aluminum/potassium perchlorate - 1 mole % silver perchlorate	60/60	6.4	6.2	0	0	0
	7. Aluminum/potassium perchlorate - 1 mole % cupric perchlorate	60/60	3.3	2.9	0	0	0
	8. Aluminum/potassium perchlorate - 1 mole % potassium iodide	60/60	6.1	4.8	0	3.3	0
March 1961	9. Calcium/aluminum/potassium perchlorate (control)	30/20/50	5.2	11.6	122.0	-	-
	10. Calcium/aluminum/potassium perchlorate 1 mole % silver perchlorate	30/20/50	8.2	16.7	79.0	58.0	27.0
	11. Calcium/aluminum/potassium perchlorate 1 mole % potassium iodide	30/20/50	4.9	10.5	121.0	0	±
March 1961	12. Aluminum/calcium fluoride/potassium perchlorate (control)	31/20/49	2.2	6.7	112.0	-	-
	13. Aluminum/calcium fluoride/potassium perchlorate - 1 mole % silver perchlorate	31/20/49	1.8	6.9	232.0	0	27.0
	14. Aluminum/calcium fluoride/potassium perchlorate - 1 mole % cupric perchlorate	31/20/49	1.8	Erratic performance		0	-
	15. Aluminum/calcium fluoride/potassium perchlorate - 1 mole % potassium iodide	31/20/49	1.9	5.3	163.0	0	6.0
DOPED PERCHLORATES PREPARED ON PLANT SCALE							
Sept 1961	16. Calcium/aluminum/potassium perchlorate (control)	30/20/50	5.9	12.5	112.0	-	-
	17. Calcium/aluminum/potassium perchlorate 1 mole % silver perchlorate	30/20/50	6.4	12.9	131.0	9.5	3.2
	18. Calcium/aluminum/potassium perchlorate 1 mole % silver perchlorate (light exposed)	30/20/50	6.4	14.0	119.0	9.5	13.7
	19. Calcium/aluminum/potassium perchlorate 2 mole % silver perchlorate	30/20/50	5.6	12.4	121.0	0	0
Sept 1961	20. Aluminum/calcium fluoride/potassium perchlorate (control)	31/20/49	2.5	5.1	155.0	-	-
	21. Aluminum/calcium fluoride/potassium perchlorate - 1 mole % silver perchlorate	31/20/49	Insufficient light to record			-	-
	22. Aluminum/calcium fluoride/potassium perchlorate - 1 mole % silver perchlorate (light exposed)	31/20/49	1.8	6.6	267.0	0	29.6
	23. Aluminum/calcium fluoride/potassium perchlorate - 2 mole % silver perchlorate	31/20/49	1.9	5.3	163.0	0	0

TABLE 5

Impact and Friction Sensitivity Data

Composition	Percent	Impact Sensitivity PA (inches)	Friction Pendulum	
			Steel	Fiber
Aluminum/potassium perchlorate	40/60	23	CB	NA
Aluminum/potassium perchlorate 1 M% silver perchlorate doped	40/60	-	-	-
Aluminum/potassium perchlorate 1 M% cupric perchlorate doped	40/60	-	-	-
Aluminum/potassium perchlorate 1 M% potassium iodide doped	40/60	-	-	-
Calcium/aluminum/potassium perchlorate	30/20/50	14	Det	NA
Calcium/aluminum/potassium perchlorate 1 M% silver perchlorate doped	30/20/50	19	CD	NA
Calcium/aluminum/potassium perchlorate 1 M% potassium iodide doped	30/20/50	19	CD	NA
Aluminum/calcium fluoride/potassium perchlorate	31/20/49	27	Det	NA
Aluminum/calcium fluoride/potassium perchlorate 1 M% silver perchlorate doped	31/20/40	24	NA	NA
Aluminum/calcium fluoride/potassium perchlorate 1 M% cupric perchlorate doped	31/20/49	28	NA	NA
Aluminum/calcium fluoride/potassium perchlorate 1 M% potassium iodide doped	31/20/49	25	Det	NA

CB = complete burning, CD = complete detonation, NA = no action, Det = detonation, and - = not available.

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Accession No. _____ AD _____
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 David J. Edelman and Seymour M. Kaye

Feltman Research Laboratories, Picatinny Arsenal
 Technical Memorandum 1091, October 1963, 20 pp., tables, figure. Unclassified report.

The effects of adding impurities such as Ag^+ , Cu^{++} , and I^- ions to the oxidant upon the luminosity of flash systems were tested in the following blends: (1) FP600 (60/40 KClO_4/Al) photoflash composition for sea level use, (2) FP790 (30/20/50 $\text{Ca}/\text{Al}/\text{KClO}_4$) photoflash composition for high altitude use, and (3) FP856 (31/20/49 $\text{Al}/\text{CaF}_2/\text{KClO}_4$) high altitude flash composition. All tests were performed at sea level or at 20.9 mm Hg, simulating 80,000 feet altitude, and at the temperature prevailing at the Pyrotechnic Laboratory High Altitude Tank.

(over)

1. Potassium perchlorate — Impurities
2. Pyrotechnics — Impurities

- I. Edelman, David J.
- II. Kaye, Seymour M.
- III. Title: Photoflash compositions

UNITERMS

Potassium perchlorate
 Photoflash powder
 Luminosity
 Impurities
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 Copper
 Iodide

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The above systems doped with Cu⁺⁺ and I⁻ ions either failed to give verifiable results or exhibited lower efficiency values. With the Cu⁺⁺ ion, an undesirable reaction between its water of hydration and the calcium occurred. Increasing the concentration of Ag⁺ ions from 1 to 2 mole percent did not improve luminous efficiencies.

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